

US EPA ARCHIVE DOCUMENT

DP Barcode :D169723, 178012  
PC Code No. :J12701  
EFGWB Out : NOV 9 1992

TO: Barbara Briscoe  
Product Manager PM 51  
Special Review and Reregistration Division (H7508W)

FROM: Akiva Abramovitch, Chief  
Environmental Chemistry Review Section #3  
Environmental Fate & Ground Water Branch/EFED (H7507C)

THRU: Henry Jacoby, Chief  
Environmental Fate & Ground Water Branch/EFED (H7507C)

*[Signature]*  
For AA

Attached, please find the EFGWB review of...

Reg./File # :2755

Common Name :Brodifacoum

Product Name :Talon, Havoc

Company Name :ICI Americas, Inc.

Purpose :Review of 161-1 and 163-1 studies

Type Product :Rodenticide days Action Code: 627 EFGWB #(s): 92-0044, 0884. Review Time: 2.0

EFGWB Guideline/MRID/Status Summary Table: The review in this package contains...

161-1	42237701	N	162-4		164-4		166-1	
161-2			163-1	42024501	N	164-5		
161-3			163-2			165-1		166-2
161-4			163-3			165-2		166-3
162-1			164-1			165-3		167-1
162-2			164-2			165-4		167-2
162-3			164-3			165-5		201-1
								202-1

Y = Acceptable (Study satisfied the Guideline)/Concur P = Partial (Study partially satisfied the Guideline, but additional information is still needed)  
S = Supplemental (Study provided useful information, but Guideline was not satisfied) N = Unacceptable (Study was rejected)/Non-Concur

DP BARCODE: D169723

REREG CASE # 2755

CASE: 816404  
SUBMISSION: S404785

DATA PACKAGE RECORD  
BEAN SHEET

DATE: 10/09/91  
Page 1 of 1

\* \* \* CASE/SUBMISSION INFORMATION \* \* \*

CASE TYPE: REREGISTRATION ACTION: 627 GENERIC DATA SUBMISSION  
CHEMICALS: 112701 Brodifacoum

ID#: 112701-010182

COMPANY: 010182 ICI AMERICAS INC

PRODUCT MANAGER: 51 BARBARA BRISCOE

703-308-8065

ROOM: CS1

3H3

PM TEAM REVIEWER: FRANKLIN RUBIS

703-308-8184

ROOM: CS1

4J6

RECEIVED DATE: 09/19/91

DUE OUT DATE: 12/18/91

\* \* \* DATA PACKAGE INFORMATION \* \* \*

DP BARCODE: 169723

EXPEDITE: N

DATE SENT: 10/09/91

DATE RET.: / /

CHEMICAL: 112701 Brodifacoum

DP TYPE: 999 Miscellaneous Data Package

ADMIN DUE DATE: 01/07/92

CSF: N

LABEL: N

ASSIGNED TO	DATE IN	DATE OUT
DIV : EFED	10/10/91	/ /
BRAN: EFGB	/ /	/ /
SECT:	/ /	/ /
REVR :	/ /	/ /
CONTR:	/ /	/ /

\* \* \* DATA REVIEW INSTRUCTIONS \* \* \*

PLEASE REVIEW THE ATTACHED DATA, GRN 163-1, ADSORPTION AND  
DESORPTION IN SOILS MEASURED UNDER LAB. CONDITONS, MRID  
420245-01

\* \* \* ADDITIONAL DATA PACKAGES FOR THIS SUBMISSION \* \* \*

DP BC	BRANCH/SECTION	DATE OUT	DUE BACK	INS	CSF	LABEL
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2

ADA  
2/8

DP Barcode : 178012  
PC Code No. :  
EFGWB Out :

TO: Product Manager PM  
Special Review and Reregistration Division (H7508W)

FROM: Akiva D. Abramovitch, Ph.D., Head  
Environmental Chemistry Review Section #3  
Environmental Fate & Ground Water Branch/EFED (H7507C)

THRU: Henry Jacoby, Chief  
Environmental Fate & Ground Water Branch/EFED (H7507C)

Attached, please find the EFGWB review of...

Reg./File # : \_\_\_\_\_

Common Name : Brodifacoum

Product Name : \_\_\_\_\_

Company Name : \_\_\_\_\_

Purpose : \_\_\_\_\_

Type Product : \_\_\_\_\_ Action Code: 627 EFGWB #(s): 92-0884 Review Time: \_\_\_\_\_ days

EFGWB Guideline/MRID/Status Summary Table: The review in this package contains...

161-1	162-4	164-4	166-1
161-2	163-1	164-5	166-2
161-3	163-2	165-1	166-3
161-4	163-3	165-2	167-1
162-1	164-1	165-3	167-2
162-2	164-2	165-4	201-1
162-3	164-3	165-5	202-1

Y = Acceptable (Study satisfied the Guideline)/Concur P = Partial (Study partially satisfied the Guideline, but additional information is still needed)  
S = Supplemental (Study provided useful information, but Guideline was not satisfied) N = Unacceptable (Study was rejected)/Non-Concur

DP BARCODE: D178012

REREG CASE # 2755

CASE: 819451  
SUBMISSION: S417480

DATA PACKAGE RECORD  
BEAN SHEET

DATE: 05/11/92  
Page 1 of 1

\* \* \* CASE/SUBMISSION INFORMATION \* \* \*

CASE TYPE: REREGISTRATION ACTION: 627 GENERIC DATA SUBMISSION  
CHEMICALS: 112701 Bromo-(1,1-biphenyl)-4-yl)-1,2,4,4-tetrahydro-1-na 100.00 %

ID#: 112701

COMPANY:

PRODUCT MANAGER: 51 BARBARA BRISCOE

PM TEAM REVIEWER: FRANKLIN RUBIS

RECEIVED DATE: 05/11/92 DUE OUT DATE: 09/08/92

703-308-8177 ROOM: CS1 3H3  
703-308-8184 ROOM: CS1 4J6

\* \* \* DATA PACKAGE INFORMATION \* \* \*

DP BARCODE: 178012 EXPEDITE: N DATE SENT: 05/11/92 DATE RET.: / /  
CHEMICAL: 112701 Bromo-(1,1-biphenyl)-4-yl)-1,2,4,4-tetrahydro-1-naphthyl)-4  
DP TYPE: 999 Miscellaneous Data Package

ADMIN DUE DATE: 09/08/92

CSF: N

LABEL: N

ASSIGNED TO	DATE IN	DATE OUT
DIV : EFED	05/19/92	/ /
BRAN: EFGB	/ /	/ /
SECT:	/ /	/ /
REVR :	/ /	/ /
CONTR:	/ /	/ /

\* \* \* DATA REVIEW INSTRUCTIONS \* \* \*

Please review MRID 422377-01 and provide me with your comments.

\* \* \* ADDITIONAL DATA PACKAGES FOR THIS SUBMISSION \* \* \*

DP BC	BRANCH/SECTION	DATE OUT	DUE BACK	INS	CSF	LABEL
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4

1. CHEMICAL: Common name:

Brodifacoum.

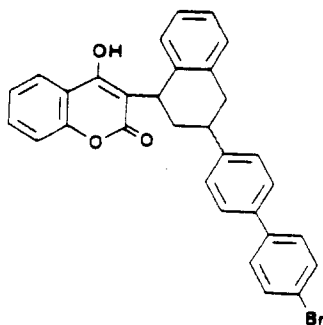
Chemical name:

3-[3-(4'-Bromo-1,1'-biphenyl-4-yl)-1,2,3,4-tetrahydro-1-naphthyl]-4-hydroxycoumarin.

Trade name(s):

Talon, Havoc.

Structure:



Formulations:

Ready-to-use grain-base in pellets, mini pellets, and wax blocks.

Physical/Chemical properties:

Molecular formula:  $C_{31}H_{23}BrO_3$ .  
Molecular weight: 523.4.  
Physical state: Off-white powder.  
Melting point: 228-232 C.  
Vapor pressure (25 C): <0.13 mPa.  
Solubility (20 C): <10 mg/L water at pH 7; 6-20 g/L acetone; <0.6-6.0 mg/L benzene; 3 g/L chloroform.

2. TEST MATERIAL:

Study 1 and 2: Active ingredient.

3. STUDY/ACTION TYPE:

Review of mobility (batch equilibrium) and hydrolysis studies.

4. STUDY IDENTIFICATION:

Jackson, R., I. Priestly, and B.E. Hall. 1991. The Determination of the Hydrolytic Stability of [<sup>14</sup>C]-Brodifacoum. Laboratory Project No. 381420. Unpublished study performed by Inveresk Research International, Tranent, Scotland, and submitted by ICI Americas Inc., Wilmington, DE. (42237701)

Newby, S.E., and B.G. White. 1979. Brodifacoum: Adsorption and desorption in soils measured under laboratory conditions. Laboratory Project ID: TMJ 1764 B. Unpublished study performed by ICI Agrochemicals, Bracknell, Berkshire, UK, and submitted by ICI Americas Inc., Wilmington, DE. (42024501)

5. REVIEWED BY:

David Edelstein  
Soil Scientist  
EFGWB/EFED/OPP  
Review Section #3

Signature: David Edelstein

Date: 10/16/92

6. APPROVED BY:

Akiva Abramovitch  
Chief  
EFGWB/EFED/OPP  
Review Section #3

Signature: Akiva Abramovitch for AA Abramovitch

Date: 10/16/92

7. CONCLUSION:

161-1: Hydrolysis (MRID 42237701; unacceptable)

These data are considered to be of uncertain value and should not be used to predict the environmental behavior of brodifacoum residues. This study is unacceptable because brodifacoum degradates were not adequately identified and evidence of storage stability was not provided although samples were stored frozen before analysis. Brodifacoum appeared to degrade rapidly for 24 hours at pH 5, 7 and 9, but no degradation was observed after that time. No explanation was offered for this unusual chemical behavior aside from the admission of possible methodology problems. It is unlikely that all of these problems can be resolved, and a new study will be required.

163-1: Mobility-Adsorption/Desorption (MRID 42024501; unacceptable)

These data are considered to be of uncertain value and should not be used to predict the environmental behavior of brodifacoum residues. This study is unacceptable because acetone was used as a co-solvent, resulting in brodifacoum concentrations far in excess of possible concentrations in the field. Brodifacoum is soluble in acetone at up to 20,000 ppm. Brodifacoum was applied to a 2 g soil/20 ml water slurry at 0.9-4.5 ppm, although the study author stated that Brodifacoum solubility in water is <0.1 ppm in 0.01 N CaCl<sub>2</sub> solution. It is not possible to extrapolate these results into realistic solubility ranges, or to discount the likelihood that Brodifacoum

was partitioned out of the aqueous solution and into the acetone co-solvent. In addition, Freundlich K values were not calculated.

8. RECOMMENDATIONS:

Inform the registrant that:

1) The Phase IV Review of brodifacoum (EFGWB #90-0881) of 11/19/90 states that the data requirements imposed for brodifacoum are hydrolysis, aerobic soil metabolism, and mobility (adsorption/desorption or leaching).

2) The hydrolysis study must be repeated with sufficient analytical rigor to identify all degradates present at concentrations greater than 10% of the applied or 0.1 ppm, whichever is smaller, as well as any change in the distribution of brodifacoum between the cis and trans isomers.

3) If batch equilibrium is performed, a preliminary study will be necessary to determine the maximum solubility of Brodifacoum in 0.01 N  $\text{CaCl}_2$  solution. Test concentrations should not exceed this maximum solubility. Studies on at least four U.S. soils are required.

4) If column leaching studies are performed, at least two, and preferably four, U.S. soils must be used. If acceptable hydrolysis and aerobic metabolism studies show that brodifacoum is unstable to abiotic hydrolysis or biodegradation, mobility data will be required for all major degradates, either as batch equilibrium with each individual degrade or as an aged soil column leaching study including at least two U.S. soils.

9. BACKGROUND:

A. Introduction

B. Directions for Use

Brodifacoum is an anticoagulant rodenticide registered for use to control Norway rats, roof rats, and house mice (including warfarin resistant strains) in public, industrial, farm, and commercial buildings. Brodifacoum may also be used in residential and urban indoor/outdoor areas by professional pest control personnel. Single active ingredient formulations include ready-to-use grain-base bait in pellets, mini pellets, and wax blocks.

10. DISCUSSION OF INDIVIDUAL TESTS OR STUDIES:

Refer to attached reviews.

11. COMPLETION OF ONE-LINER:

12. CBI APPENDIX:

All data reviewed here are considered "company confidential" by the registrant and must be treated as such.



# **BRODIFACOU**

## **TASK 1: REVIEW AND EVALUATION OF INDIVIDUAL STUDIES**

July 27, 1992

Final Report.

BRODIFACOUM

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Scientific Studies	
1. Hydrolysis (Jackson, Priestly and Hall, 42237701)	1.1
2. Mobility (batch equilibrium). (Newby and White, 42024501)	2.1
References	3.1
Appendix	3.2

## INTRODUCTION

Brodifacoum is an anticoagulant rodenticide registered for use to control Norway rats, roof rats, and house mice (including warfarin resistant strains) in public, industrial, farm, and commercial buildings. Brodifacoum may also be used in residential and urban indoor/outdoor areas by professional pest control personnel. Single active ingredient formulations include ready-to-use grain-base bait in pellets, mini pellets, and wax blocks.



DATA EVALUATION RECORD

STUDY 1

CHEM 112701

Brodifacoum

§161-1

FORMULATION--00-ACTIVE INGREDIENT

STUDY ID 42237701

Jackson, R., I. Priestly, and B.E. Hall. 1991. The Determination of the Hydrolytic Stability of [ $^{14}\text{C}$ ]-Brodifacoum. Laboratory Project No. 381420. Unpublished study performed by Inveresk Research International, Tranent, Scotland, and submitted by ICI Americas Inc., Wilmington, DE. (42237701)

DIRECT REVIEW TIME = 8

REVIEWED BY: D. Edelstein  
TITLE: Soil Scientist  
ORG: EFGWB/EFED/OPP  
TEL: 703-305-5463

SIGNATURE:

*D. Edelstein* 10/16/92

CONCLUSIONS:

Hydrolysis

1. This study cannot be used to fulfill data requirements.
2. These data are considered to be of uncertain value and should not be used to predict the environmental behavior of brodifacoum residues.
3. This study is unacceptable for the following reason:
  - brodifacoum degradates were not adequately identified.
  - no explanation was offered for the unusual degradation kinetics of brodifacoum in water.
  - evidence of storage stability was not provided; samples were stored frozen before analysis.
4. Since it is not likely that degradates from this study are available for further analysis, this study cannot be resolved with the submission of additional data. Therefore, a new study must be submitted. If samples are stored, storage stability data must be provided. If unusual degradation patterns are observed, an explanation is required.

## METHODOLOGY:

[<sup>14</sup>C]-brodifacoum was repurified by TLC to radiopurity of 97.91% with a cis:trans isomer ratio of 58.30:41.70. 100  $\mu$ l aliquots of [<sup>14</sup>C]-brodifacoum in acetonitrile were tested in buffered solutions at pH 5, 7, and 9 to determine the solubility of [<sup>14</sup>C]-brodifacoum in buffered aqueous solution. At pH 5, at a target concentration of 0.05  $\mu$ g/ml in the presence of 0.4% (by volume) acetonitrile, 94% of the radioactivity was in solution. All of the radioactivity was in solution at these concentrations at pH 7 and 9. No brodifacoum adsorbed to the glass flasks. Based on these observations, a target concentration of 0.04  $\mu$ g/ml brodifacoum with 0.8% acetonitrile by volume as cosolvent was chosen.

Aliquots of 0.8 ml of 5.00  $\mu$ g/ml [<sup>14</sup>C]-brodifacoum solution were added to 100 ml of sterile buffer solution to give a nominal concentration of 0.04  $\mu$ g/ml. Zero-time test solutions were prepared after preparation of all other solutions to allow immediate analysis to be carried out. The total amount of [<sup>14</sup>C]-brodifacoum added to zero-time flasks was calculated as 4.59  $\mu$ g/ml.

The flasks containing the test solutions were incubated at  $25 \pm 1$  C in the dark for up to thirty days. Duplicate 1 gram samples were taken at the following intervals after application: 0, 1, 3, 7, 14, 21, and 30 days. All incubates were stored at ca -20 C prior to extraction.

Sodium chloride (5 g) was added to each test solution and the solution was adjusted to pH 2 with 1 M HCl. Each sample was partitioned twice with dichloromethane and the extracts combined. Each incubation flask was washed with acetone. The total volumes of dichloromethane, aqueous and acetone fractions were recorded and aliquots of each (2 x 0.5 ml) were assayed by LSC. A fixed proportion of each organic extract and acetone flask wash was pooled and concentrated to a small volume under a stream of nitrogen.

Samples of each concentrated pooled extract were analyzed by TLC on silica gel using the following solvent systems:

System 1: Chloroform (100%)

System 3: Dioxan:petroleum ether (3:7 v:v)

Non-radiolabelled brodifacoum and 4-hydroxycoumarin were co-chromatographed with each sample extract. Following chromatography, radioactivity on TLC plates was quantified using a RITA 68000 linear analyzer.

Zero-time and Day 30 samples were analyzed by HPLC. A Hewlett-Packard 1050 system was connected to an Apex Silica 5  $\mu$ m column and a Bethold LB 507A Radiodetector. The mobile phase was hexane:dichloromethane:acetic acid (75:25:0.6, v:v:v) at a flow rate of 1 ml/min. Ultraviolet detection was carried out at 254 nm. The radioactivity in 15 s fractions of column eluate was determined by

LSC. Brodifacoum and 4-hydroxycoumarin were chromatographed as reference standards.

Radioactivity was analyzed using a liquid scintillation analyzer with automatic quench correction by external standard-channels ratio. Each individual sample was counted for 5 min. or for the time taken to detect 900,000 counts. A background count rate was determined using scintillation fluid containing blank sample matrix which was subtracted from each sample count rate. Data reliability limit was 30 d.p.m. above background.

#### DATA SUMMARY:

No conclusions could be drawn concerning the hydrolysis of [<sup>14</sup>C]brodifacoum because freezer storage stability of brodifacoum was not demonstrated. Also, a preliminary experiment indicated that brodifacoum at the test concentration would not precipitate or adhere to the glass test flasks. In the actual study, radioactivity decreases in solution of 10% at pH 7 and 60% at pH 5 were attributed to precipitation and/or adsorption to glass.

Chromatographic analysis of the test solutions indicated that [<sup>14</sup>C]brodifacoum degraded over the first 24 hr at all three pH values, but that degradation did not occur over the remainder of the 30 day study. No hydrolytic half life could be calculated on the basis of this degradation period. Degradation was greatest at pH 9 (40%) and least at pH 5 (25%). A highly polar material was found near the origin of the TLC system may have consisted of more than one component including 4-hydroxycoumarin.

#### COMMENTS:

1. The study author states, "Degradation observed at Day 1 and at all subsequent analysis points may be due to methodology problems." No physical explanation is offered for the apparent early degradation of brodifacoum followed by an indefinite period of hydrolytic stability. The registrant should explain these unusual reaction kinetics.
2. All degradates greater than 10% of the applied or 0.1 ppm should be identified. The TLC and HPLC systems employed here did not provide sufficient resolution to identify degradates. Analytical standards other than 4-hydroxycoumarin may have to be employed in order to fully characterize brodifacoum hydrolytes.
3. When study samples are stored, data must be provided demonstrating that the study results will not be affected by storage. No evidence was presented here to indicate that frozen storage of study samples might not compromise sample integrity.
4. Recoveries of radioactivity at the three test pH levels ranged from 82.88% to 108.06%.

STUDY AUTHOR(S)'S RESULTS AND/OR CONCLUSIONS  
(INCLUDING PERTINENT TABLES AND FIGURES)



5. The study author suggests that brodifacoum was lost from solution by precipitation and adsorption to glass. However, this did not occur in the preliminary study of appropriate brodifacoum concentrations for the experiment. No explanation is offered for this inconsistency.

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Page \_\_\_\_\_ is not included in this copy.

Pages 17 through 38 are not included in this copy.

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The material not included contains the following type of information:

- \_\_\_\_\_ Identity of product inert ingredients.
- \_\_\_\_\_ Identity of product impurities.
- \_\_\_\_\_ Description of the product manufacturing process.
- \_\_\_\_\_ Description of quality control procedures.
- \_\_\_\_\_ Identity of the source of product ingredients.
- \_\_\_\_\_ Sales or other commercial/financial information.
- \_\_\_\_\_ A draft product label.
- \_\_\_\_\_ The product confidential statement of formula.
- \_\_\_\_\_ Information about a pending registration action.
- ☒ FIFRA registration data.
- \_\_\_\_\_ The document is a duplicate of page(s) \_\_\_\_\_.
- \_\_\_\_\_ The document is not responsive to the request.
- \_\_\_\_\_ Internal deliberative information.
- \_\_\_\_\_ Attorney-Client work product.
- \_\_\_\_\_ Claimed Confidential by submitter upon submission to the Agency.

The information not included is generally considered confidential by product registrants. If you have any questions, please contact the individual who prepared the response to your request.

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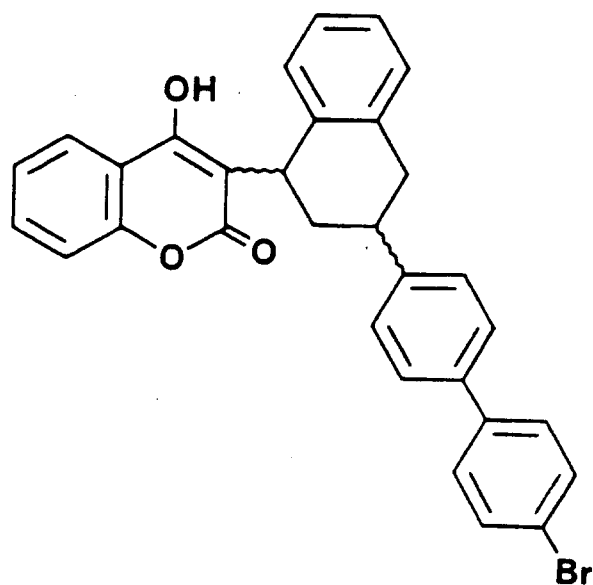
## REFERENCES

The following studies were reviewed:

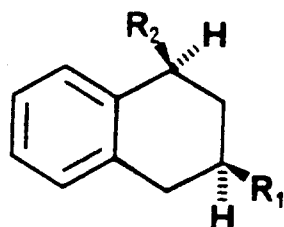
Jackson, R., I. Priestly, and B.E. Hall. 1991. The Determination of the Hydrolytic Stability of [ $^{14}\text{C}$ ]-Brodifacoum. Laboratory Project No. 381420. Unpublished study performed by Inveresk Research International, Tranent, Scotland, and submitted by ICI Americas Inc., Wilmington, DE. (42237701)

Newby, S.E., and B.G. White. 1979. Brodifacoum: Adsorption and desorption in soils measured under laboratory conditions. Laboratory Project ID: TMJ 1764 B. Unpublished study performed by ICI Agrochemicals, Bracknell, Berkshire, UK, and submitted by ICI Americas Inc., Wilmington, DE. (42024501)

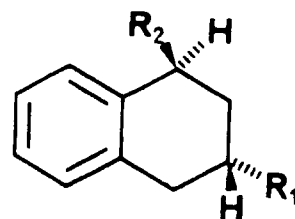
APPENDIX  
BRODIFACOUM



3-[3-(4'-Bromo-1,1'-biphenyl-4-yl)-1,2,3,4-tetrahydro-1-naphthyl]-4-hydroxycoumarin  
(Brodifacoum)



Cis Isomer



Trans Isomer



DATA EVALUATION RECORD

STUDY 2

CHEM 112701

Brodifacoum

§163-1

FORMULATION--00-ACTIVE INGREDIENT

STUDY ID 42024501

Newby, S.E., and B.G. White. 1979. Brodifacoum: Adsorption and desorption in soils measured under laboratory conditions. Laboratory Project ID: TMJ 1764 B. Unpublished study performed by ICI Agrochemicals, Bracknell, Berkshire, UK, and submitted by ICI Americas Inc., Wilmington, DE.

DIRECT REVIEW TIME = 20

REVIEWED BY: N. Shishkoff  
TITLE: Staff Scientist  
ORG: Dynamac Corporation  
Rockville, MD  
TEL: 301-417-9800

APPROVED BY: D. Edelstein  
TITLE: Soil Scientist  
ORG: EFGWB/EFED/OPP  
TEL: 703-305-5463

SIGNATURE:

*D. Edelstein* 10/16/92

CONCLUSIONS:

Mobility - Leaching and Adsorption/Desorption

1. This study cannot be used to fulfill data requirements.
2. These data are considered to be of uncertain value and should not be used to predict the environmental behavior of brodifacoum residues.
3. This study is unacceptable for the following reason:  
A co-solvent was used, resulting in unrealistically high test concentrations of brodifacoum.  
Freundlich K values were not calculated.
4. Since the use of a co-solvent with high concentrations of brodifacoum does not describe actual brodifacoum mobility in the soil solution,

the problems with this study cannot be resolved with the submission of additional data. Therefore, a new study must be submitted.

#### METHODOLOGY:

Sandy clay loam, loam, and sand soils (Table 2) were sieved (2 mm) and air-dried. Subsamples of the soil were further sieved (1 mm) and mixed with 0.01 M calcium chloride solution. The soil:calcium chloride solution slurries (approximately 2 g:20 mL) were shaken overnight. After equilibration, a cis:trans isomer mixture (64:36) of ring-labeled [ $^{14}\text{C}$ ]brodifacoum (radiochemical purity approximately 93%, specific activity 14.75 mCi/mMol, ICI; uniformly labeled in the phenyl ring of the coumarin moiety), dissolved in acetone, was added to the slurries at 0.9, 1.8, 2.7, 3.6, and 4.5 ppm. The soil:calcium chloride solution slurries were vortexed for 1 minute, then equilibrated on a shaker for 16-20 hours at 21-22 C in the dark. After the equilibration period, the slurries were centrifuged and aliquots (1 mL) of the supernatants were removed and analyzed by LSC.

To determine the desorption of brodifacoum, an additional aliquot (2 mL) of the supernatant was removed, and the total volume of the removed supernatant (3 mL) was replaced with pesticide-free 0.01 M calcium chloride solution. The slurries were reequilibrated for 16-20 hours, centrifuged, and aliquots (1 mL) of the supernatants were removed and analyzed by LSC. Additional aliquots (3 mL) of the supernatants were removed, the total volume of the removed supernatant (4 mL) was replaced with pesticide-free 0.01 M calcium chloride solution, and the slurries were reequilibrated for 16-20 hours. The desorption step was repeated two more times, with a total of 5 mL and 6 mL being removed after the equilibration periods (Table 3).

Following desorption, aliquots from the supernatants of the 4.5 ppm brodifacoum test solutions were partitioned twice with methylene chloride. The aqueous phases were combined and aliquots were analyzed by LSC; the remaining solution was concentrated by evaporation and aliquots were analyzed by TLC on silica gel plates developed in dioxane:petroleum ether (30:70) or chloroform:methanol:ethyl acetate (60:30:10). Radioactive areas were visualized by autoradiography and were quantified by radioscanning and by scraping the radioactive area from the plates and analyzing by LSC following combustion. Reference compounds were cochromatographed and visualized by UV fluorescence quenching. Aliquots of the methylene chloride extracts were analyzed by TLC as described above.

Following desorption, the soils from slurries treated with brodifacoum at 4.5 ppm were extracted twice with hexane:acetone (1:1) for 18 hours with shaking. After each extraction, the slurry was centrifuged, the supernatant was decanted, and the soil was washed with additional solvent. The extracts and soil rinsates were combined and allowed to evaporate to dryness at room temperature.



The residues were redissolved in acetone and aliquots were analyzed by TLC as previously described. Subsamples of the extracted soil were analyzed by LSC following combustion.

A similar experiment was conducted with soil slurries prepared as previously described and treated at approximately 6 ppm with either the cis or trans brodifacoum isomer dissolved in methylene chloride. These slurries apparently were equilibrated for adsorption and desorption studies as previously described.

An experiment was conducted to determine the solubility of brodifacoum in calcium chloride solution. Brodifacoum (0.1-10 ug) in an unspecified organic solvent was added to tubes, the solvent was evaporated, and 20 mL of 0.01 M calcium chloride solution were added. The tubes were sealed and the solution was sonicated for 24 hours, then aliquots of the solution were removed and analyzed by LSC. Due to the insolubility of brodifacoum, a series of sequential procedures was attempted to increase the solubility, including additional shaking, addition of detergent, and filtration (Table 14).

#### DATA SUMMARY:

No conclusions could be drawn concerning the adsorption/desorption of [<sup>14</sup>C]brodifacoum in soil:0.01 M calcium chloride solution slurries (2 g:20 mL) because brodifacoum was not in solution at any of the test concentrations (0.9-4.5 ppm). A separate solubility experiment indicated that the solubility of brodifacoum was <0.1 ppm.

There appeared to be some isomerization in the soil; the initial 64:36 cis:trans mixtures were converted to 45:55 cis:trans mixtures in the sand soil, 68:32 cis:trans mixtures in the sandy clay loam soil, and 56:44 cis:trans mixtures in the loam soil (Table 13). When each isomer was added individually to the soils, some isomeric rearrangement was seen, especially in the sand soil.

#### COMMENTS:

1. Acetone was used as a co-solvent was used, and Brodifacoum concentrations were artificially high. Brodifacoum is soluble in Acetone at up to 20,000 ppm. It is impossible to extrapolate the results of this study to natural soil-water concentrations and adsorption/desorption at such concentrations. The study authors noted that most samples were "exhibiting further adsorption during the desorption phase", a possible sign of precipitation rather than adsorption. The study authors determined the solubility of brodifacoum in 0.01 M calcium chloride solution to be <0.01 ug/mL; in other literature, brodifacoum is reported to be insoluble in water (Meister, R.T. 1990. Farm Chemicals Handbook '90. Meister Publishing Co., Willoughby, Ohio). Concentrations used in this experiment ranged from 0.9-4.5 ppm.

2.  $K_d$  values were calculated, but required Freundlich K values were not.
3. The soil was too finely sieved (1 mm), so that a significant portion of the sand fraction may have been removed. Since the soil was sieved after it had been divided into 2-g subsamples, the weight of soil added to the slurry was approximate. The study authors reported that "In practice, little soil was discarded."
4. The calcareous sandy loam used in the experiments was classified as a loam soil according to the USDA Soil Classification System. It was unclear whether the samples were sieved through 1-mm mesh prior to characterization.
5. The figures were poorly explained, and the lack of descriptive legends for many of the figures made interpretation difficult. Furthermore, the study authors did not explain how the  $K_{ads}$  values were calculated.

STUDY AUTHOR(S)'S RESULTS AND/OR CONCLUSIONS  
(INCLUDING PERTINENT TABLES AND FIGURES)

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